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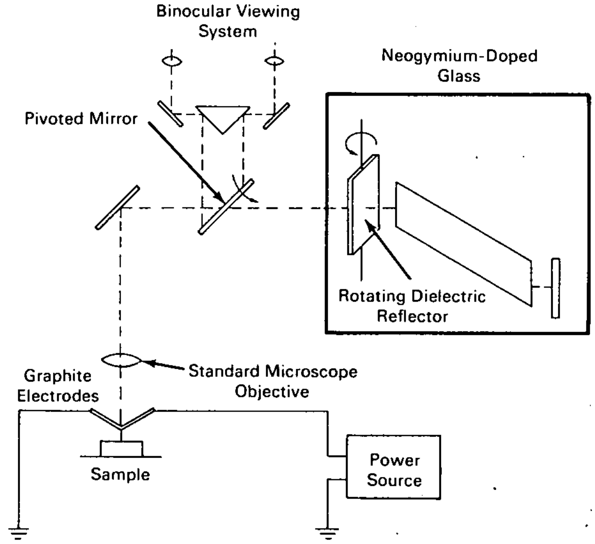
Laser Microprobe Facility Used in the Elemental Analysis of Small Feature of a Sample



SCHEMATIC DIAGRAM OF THE LASER PROBE

The problem:

To develop a laser probe facility for analyzing small areas of heterogeneous samples so that the capabilities of such a facility can be determined. The areas of major concern in laser probe analysis are: 1) the number of elements that can be analyzed; 2) the types of samples amenable to analysis; 3) spatial resolution; and 4) sensitivity.

The solution:

A laser microprobe facility has proved effective in the elemental analysis of small areas of heterogeneous samples. The instrument uses the focused beam of a pulsed laser to evaporate a small volume of material from a relatively massive sample. The vapor so produced is excited to emission of optical radiation characteristic of the elemental composition of the selected area of sample. This radiation is analyzed by conventional emission spectroscopic method to produce qualitative and/or quantitative information about the composition of chosen region of the sample.

Any element that can be analyzed by conventional dc arc emission spectroscopy can be analyzed by the laser probe technique. This includes 60 to 70 elements.

The laser probe can analyze a huge range of sample types. Good electrical conductivity and surface flatness are not prerequisites. The analysis can be performed

(continued overleaf)

in air or other atmosphere; a vacuum is not required. Any shape or size of sample that can be placed on the microscope stage can be examined. Little or no sample preparation is necessary.

The spatial resolution attainable with the laser probe is a function of laser power, thermal properties of the sample and quality of the microscope objective. Resolution of one to three microns has been attained. The sensitivity is a function of laser power.

How it's done:

The laser probe components are illustrated in the figure. The active medium of the laser is a neogymium-doped glass. The laser is Q-switched by a rotating dielectric reflector. The output of the laser is a single pulse of about 10μ sec duration, or a train of several such pulses, depending upon the operating conditions. The beam is focused by a standard microscope objective on the surface of a sample. A binocular viewing system is made confocal with the laser so that microscopic selection of the area to be analyzed is possible. A pivoted mirror allows one to switch rapidly from the viewing position to the sampling position. The sample stage is movable in three dimensions.

Impact of the focused laser beam on a sample produces heating, melting and vaporization of the selected portion of sample. As little as a few tenths of a microgram of sample can be evaporated per laser pulse.

If desired, a high potential of up to two kilovolts from a power source can be applied to a set of graphite electrodes to supply additional excitation to the sample. The partially ionized vapor short circuits the electrodes, causing the power supply to discharge. This cross-excitation provides greater sensitivity than is available from the laser output alone.

The laser probe has been applied to: a) examination of a weld on an Inconel tube to determine the weld material; b) examination of corrosion deposits on SPERT fuel rod cladding; c) examination of irradiated (2.1 x 10^{22}nvt) stainless steel for metallurgical effects; and d) analysis of successive layers of deposits on metals.

Notes:

1. Complete details are available in Idaho Nuclear Corporation Report CI-1121, dated September 1966, "The Analytical Chemistry Branch Laser Microprobe Facility for the Elemental Analysis of Small Features of a Sample" by J. M. Baldwin.
2. Inquiries may be directed to:

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Patent status:

Inquiries concerning rights for commercial use of this innovation may be made to:

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